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PROJECT FINAL REPORT

Instructions:

- **Please note that making changes to the project without prior written consent from the funder(s) could constitute sufficient grounds for termination of funding.**
- This report must be a stand-alone report, *i.e.*, must be complete in and of itself. Scientific articles or other publications cannot be substituted for the report.
- A signed electronic copy of this report must be forwarded to the funders' representative on or before the due date, as per the investment agreement.
- A detailed, signed statement of revenues received and expenses incurred during the entire funding period of the project must be submitted along with this report, as per the investment agreement.
- For any questions regarding the preparation and submission of this report, please contact the funders' representative.

Section A: Project overview

1. Project number: 2017F077R

2. Project title: A route to multi-product oleochemical production in Alberta

3. Abbreviations: Define ALL abbreviations used.

LCG – Lipid Chemistry Group

UofA – University of Alberta

AFNS - Agricultural, Food and Nutritional Science

CO – Canola Oil

ECO – Epoxidized Canola Oil LO – Linseed Oil ELO – Epoxidized Linseed (Flax) Oil DEA – Diethanolamine DAG – Diacylglycerol MAG – Monoacylglycerol SSS – Tristearin CODEAm – Canola Oil Diethanolamides ECODEAm – Epoxidized Canola Oil Diethanolamides ELODEAm – Epoxidized Linseed Oil Diethanolamides ATR-FTIR – Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy RT - Retention Time OOC – Oxirane Oxygen Content [%] OHV – Hydroxyl Value [mg KOH/g]	
4. Project start date: 2017/03/01	
5. Project completion date: 2020/02/28	
6. Final report submission date: 2020/07/10	
7. Research and development team data	
a) Principal Investigator: (Requires personal data sheet (refer to Section 14) only if Principal Investigator has changed since last report.)	
Name	Institution
Jonathan Curtis	LCG, Department of AFNS, University of Alberta
b) Research team members (List all team members. For each new team member, <i>i.e.</i> , joined since the last report, include a personal data sheet. Additional rows may be added if necessary.)	
Name	Institution
Tolibjon Omonov	LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Ereddad Kharraz	LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Vinay Patel	LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta

Section B: Non-technical summary (max 1 page)

Provide a summary of the project results which could be used by the funders for communication to industry stakeholders (*e.g.*, producers, processors, retailers, extension personnel, etc.) and/or the general public. This summary should give a brief background as to why the project was carried out, what were the principal outcomes and key messages, how these outcomes and key messages will advance the agriculture industry, how they will impact industry stakeholders and/or consumers, and what are the economic benefits for the industry. This summary should be in plain, non-scientific language.

Plant oils such as canola, hemp, flax and camelina can be used as raw materials for producing a wide variety of renewable chemicals or oleochemicals. Local oleochemical production could

help the oilseed industry through access to more diverse markets. Plant-based oleochemicals would also typically have significantly lower carbon footprints compared to petrochemicals, so may provide additional environmental benefits. The University of Alberta Lipid Chemistry Group (LCG) has developed and patented several processes for the production of oleochemicals, especially for use in materials. These include biobased polyols, which can be used in the production of polymers such as polyurethane, and biobased epoxy resins that can be applied to the production of composite materials, such as those comprised of wood or plant fibres and a resin. The oleochemical industry is a multi-billion dollar industry largely based around palm and soybean oils, with many well-known products including detergents, many personal care products, and paints. The growth of oleochemical industries based around canola and other prairie- oilseeds could greatly benefit this region. Oleochemical plants typically produce a wide range of raw materials (eg fatty acids, soaps, biodiesel, fatty amides, waxes etc) which allows for great economies of scale. Likewise, this project investigates how several oleochemicals could be produced through the implementation of recently patented, multi-step process for biopolyol production, in a single plant. To do this, a pilot-scale reactor was used, and conditions were optimised for polyol production, especially for use in polyurethanes. Additionally, oleochemicals were developed and optimised, for use in a range of other industries. Follow-on projects are already underway applying these to hemp biocomposite materials used in sporting goods, and to biobased erosion control products.

Section C: Project details

1. Background (max 1 page)

Describe the project background and include the relevant scientific and development work providing the impetus for the current project.

LCG has developed plant oil based chemicals (polyols) which are used in the production of polyurethane (PU) foam insulation. We have also created resins from plant oils that will be applied to the production of biocomposite materials. These are examples of oleochemicals.

The 1st generation biopolyols had relatively high viscosity and a wide molecular weight range. To overcome this, we developed 2nd generation polyols using a different form of the fatty acids. These are low viscosity (<1.0 Pa.s) polyols with narrow MW distribution and OHV of ~270 mg KOH/g when using canola oil. The process for making 1st and 2nd generation polyols was patented and has been licensed [1]. A facility is operational (Meadow Polymers, BC) and can produce polyols for PU foam insulation and coatings. However, these 1st and 2nd generation polyols have limitations in some applications, such as when higher hydroxyl functionality and reactivity is required. Recently, LCG was granted new patent for 3rd generation polyols (3GP) [2], which have 30% higher hydroxyl numbers (>370 mg KOH/g from canola) compared to the previous generation, still moderate viscosity and a narrower range of molecular weights and

structures. 3GP has an advantage in manufacturing strong and hard PU materials such as rigid foams or coatings. Lab scale manufacturing of 3GP polyols and evaluation of these polyols in making variety of PU materials has been successful. Advantageously, this reaction sequence allows for the production of multiple types of oleochemicals from a single facility.

This project will demonstrate the manufacturing feasibility of the new generations of biopolyols at pilot scale and explore the related biobased chemical products and intermediates that can be derived from each step. This greatly adds value to the polyol process since a single plant will be able to manufacture a range of oleochemicals from abundantly available canola oil or other prairie oils. Such a plant would have the flexibility to manufacture a range of products and the ability to adapt to demand, thereby increasing the potential return on investment. This concept is already used in palm oil processing facilities which produce many oleochemicals in addition to edible oils [3].

Along with pilot scale manufacturing of new generation polyols, we will identify other potential product streams (described below) that can be made in the same facility and find use in variety of commercial applications [4]. For example, vegetable oil based polyols are used in manufacturing PU coatings, adhesives and foams. Their epoxides [5] are used in making biobased thermoset resins used as a bonding adhesive in making biobased composites from lignocellulosic fibers and forestry products, glass, carbon and other fibers [6]. We have shown that castor oil replacement polyols produced from prairie grown oils [7] can be used as drop-in replacement of castor oil in certain applications such in PU, lubricant, and personal care applications. Overall, producing oleochemicals in Alberta could greatly add value to the local economy by transforming locally grown plant oils into higher value renewable chemicals and materials.

The project will demonstrate how several oleochemicals could be produced by this process in a single plant. Applications of this work include polyurethane foams and coatings, fire retardants, building materials, other polymers and personal care ingredients.

This project will establish base processes for a multi-product oleochemical production facility in Alberta. It will demonstrate the manufacturing feasibility of new generations of polyols from canola and other plant oils at pilot scale. These expand the range of applications for biobased polyols. In the polyol manufacturing process, valuable biobased chemicals can be derived from each step. This is advantageous since it adds value and production flexibility in making a range of oleochemicals.

2. Objectives and deliverables (max 1 page)

State what the original objective(s) and expected deliverable(s) of the project were. Also describe any modifications to the objective(s) and deliverable(s) which occurred over the course of the project.

The overall objective of this project is to establish the base processes for a multi-product oleochemical production facility for Alberta. This involves using our recently proposed step-wise chemical route to convert unsaturated plant oils, primarily canola oil, into a new generation of biobased polyols with high hydroxyl functionality and high reactivity.

The two specific objectives are:

1. Demonstrating the manufacturing feasibility of new generations of polyols from canola and other plant oils at pilot scale. This involves refining the processes/products and

establishing scalable unit operations. The deliverables will be a biobased polyols covering a significantly expanded range of applications compared to current versions and a process for their commercial production.

2. Demonstrating how several oleochemicals (polyols derivatives, epoxides or derivatives, intermediate chemicals) could be produced by this process in a single plant.

3. Research design and methodology (max 4 pages)

Describe and summarise the project design, methodology and methods of laboratory/field and statistical analysis that were actually used to carry out the project. Please provide sufficient detail to determine the experimental and statistical validity of the work and give reference to relevant literature where appropriate. For ease of evaluation, please structure this section according to the objectives cited above.

(Taken from the proposal; includes all years)

This proposal will develop and produce 3GP polyols and other oleochemical products at pilot scale. To do this, the 3GP process will be converted from a lab scale demonstration into an efficient and low cost process that can be scaled to an industrial level.

Making 3GP polyol is a 3-step process as described in our patent application [2]:

- A. Conversion of unsaturated plant oils into epoxides
- B. Formation of hydroxyalkyl esters of epoxidized fatty acids (EHAЕ) via transesterification of the epoxidized oils with polyhydric alcohols
- C. Transformation of the epoxy groups of EHAЕ into hydroxyl functions via ring opening by mono- and poly-hydric alcohols

Pilot scale manufacturing will include optimization of each steps at 10-100 kg scale mainly using canola oil but also flax, hemp and camelina oils. *The intermediates produced can also provide feedstock for other oleochemical derivatives.*

A. Epoxidation:

- Epoxidation will be carried out using in-situ generated performic acid from a hydrogen peroxide and formic acid solution. This uses renewable chemicals and low temperature reactions but is slow, not all fatty acid forms are as efficiently epoxidized as we described previously and there are challenges with peroxides at large scale.
- The theoretical maximum epoxy content (oxirane oxygen content, OOC) of each vegetable oil will be calculated based on their fatty acid profiles and lipid form. Then, the maximum experimental OOC of the epoxides will be achieved by controlling the epoxidation conditions, including reaction temperature, mixing conditions, ratio of oxidizing components.
- Partially epoxidized vegetable oils will also be produced with controlled OOC, initially with the aim to use in manufacturing of castor oil replacement polyols and for cosmetic applications. The OOC will of these epoxides will be varied to achieve controllable viscosity, hydroxyl functionality and emulsification properties of the polyol or derivative after hydroxylation. Blends of oils will be used to generate the ideal fatty acid profile for each application.
- Epoxides of fatty acid methyl esters will also be produced since this provides an alternative readily available starting material with much lower viscosity and is a useful

experimental comparison. In this case canola FAME will be used to produce fully epoxidized FAMES.

These epoxidized products will be used in:

- a) producing hydroxyalkyl esters of epoxidized fatty acids (all fully epoxidized vegetable oils). Further these products will be used to produce 3GP
- b) producing castor oil replacement polyols with variety hydroxyl functionality and viscosity (mainly partially epoxidized camelina oil)
- c) producing polyols with high molecular weight and low hydroxyl functionality for flexible PU foam applications (all fully epoxidized vegetable oils)
- d) producing 2nd generation polyols
- e) making biobased epoxy resins and biocomposites (all fully epoxidized vegetable oils)

B. Transesterification:

Transesterification of the fully epoxidized oil with diols (or higher alcohols) will be carried out using anhydrous sodium methoxide. This process will achieve complete transesterification of epoxidized TAG, liberating glycerol. It is important that epoxides remain intact during this process and this will be monitored via FTIR and wet chemical methods for measuring OOC. Currently, this step is being carried out using solvent due to the insolubility of the sodium methoxide in epoxidized oil and dialcohols. Even though the solvent can be fully recovered, ideally solvent free conditions would be used with minimal catalyst residue in the product and the minimum amount of product washing and clean-up. It is necessary to consider the cost vs. benefit for using immobilized catalysts including their reusability.

For optimization of the transesterification process, catalyst and conversion to solvent free 3 transesterifications conditions will be evaluated before scale-up:

- a) use of a solvent (typically acetone) in the transesterification of epoxidized TAG oils by diols followed by aqueous neutralization/work-up to remove excess diol, liberated glycerol and sodium methoxide.
- b) acid neutralization of the product followed by evaporation/recovery of the solvent, leaving glycerol and excess diol in the transesterified product. Two neutralization procedures will be compared: 1) addition of homogenous (liquid) acid; 2) treatment of the reactive solution with heterogeneous (immobilized) acid catalyst. Regeneration of the immobilized acid catalyst will also be investigated
- c) use of strongly basic, anionic solid catalyst for the transesterification of oil epoxides by diols, either with or without solvent. This will be followed by filtration of catalyst and removal of solvent if used. Regeneration of the immobilized base catalyst will also be investigated.

- several biobased polyhydric alcohols will be used to vary chain length and functionality (primary vs secondary-OH group) whilst minimizing loss of epoxides.

C. Hydroxylation (polyols):

3GP polyol: will be produced using transesterified products of epoxidized fatty acids through hydroxylation reactions via epoxy ring opening with diols in that presence of acidic catalyst, without use of solvent.

- strongly acidic solid catalyst of the Amberlyst series will be used for hydroxylation via epoxy ring opening
- 2 or more biobased dialcohols to be used for epoxy ring opening to vary chain length and functionality (primary vs secondary-OH group), whilst minimizing the degree of oligomerization.
- Work-up of the final polyols will be carried out in 2 different ways depending on transesterification path used:
 - a) filtration of solid catalyst followed by aqueous work-up of polyols to remove excess of diol
 - b) filtration of solid catalyst followed by removal of excess diol using wiped film evaporator (WFE) polyols will be evaluated in multiple PU applications with initial emphasis on rigid foams and coatings

Castor oil replacement polyol: Pilot scale castor oil replacement polyols will be produced from partially epoxidized vegetable oils or blends including camelina oil, via epoxy ring opening reactions with monoalcohols. The viscosity and hydroxyl functionality of these polyols can be manipulated via these reactions as we have described [8].

Strongly acidic solid Amberlyst catalysts will be used for hydroxylation.

Work-up of polyols will be carried out via filtration of the solid catalyst followed by removal of excess alcohols by distillation at low atmospheric pressure. This polyol can be used as drop-in replacement of castor oil in several commercial applications such as in coatings and cosmetics.

Polyols for flexible foam applications: High molecular weight polyols for flexible foam applications will be produced using epoxidized vegetable oils via epoxy ring opening process using unsaturated fatty acids produced by saponification of vegetable oils at high temperatures. The viscosity and molecular weight of the final polyol will be varied via controlling the epoxy content of the epoxidized oil and amount of unsaturation of FFA mixtures. The work-up of polyols will be carried out via removal of excess FFA using wiped film evaporator (WFE) at low atmospheric pressure. This polyol is to be used in producing flexible PU foams.

Epoxy resins: Fully epoxidized vegetable oils (canola, hemp, flax) and their derivatives from pilot scale manufacturing, specifically from hemp and flax oils, will be used for producing biobased epoxy resin and their composites with lignocellulosic fibers. Various curing agents will be used to cure biobased epoxy resins in other projects. Here, derivatization of the epoxides and transesterified epoxies will be investigated as potential alternative curing agents.

Other possible manufacturing capabilities: Fully epoxidized vegetable oils and hydroxylated epoxides of fatty acids will also be used as reactive intermediates that can be converted to other monomers. These products can be used as reactive precursor in variety of applications including:

- a) acrylated-epoxidized oils for UV curing to make thermoset resins;
- b) polyols made via epoxy ring opening using amino alcohols for self-catalyzing precursor,
- c) polyols with enhanced fire retardancy made using siloxanes and/or phosphates;
- d) polyols of varying structure can be produced and further reacted to form longer chain dimeric or oligomeric structures for flexible foams or elastomers.

4. Results, discussion and conclusions (max 8 pages)

Present the project results and discuss their implications. Discuss any variance between expected targets and those achieved. Highlight the innovative, unique nature of the new knowledge generated. Describe implications of this knowledge for the advancement of agricultural science. For ease of evaluation, please structure this section according to the objectives cited above.

NB: Tables, graphs, manuscripts, etc., may be included as appendices to this report.

Summary of the activities described in detail in the 2017/2018 interim report:

1. Optimization of epoxidation reaction at >20kg scale for canola, flax, camelina and hemp oils.
2. Pilot and lab scale production of 3rd generation polyols with studies step 2: transesterification of epoxidized plant oil (with diols); and step 3: hydroxylation of the transesterified derivatives of the epoxidized fatty acids with diols via epoxy ring opening.
3. Evaluation of 3rd generation polyols in polyurethanes.
4. Further use of the pilot scale intermediates in other products/projects including developing a fire-retardant polyol (Alberta Innovates project); making bioresins for biocomposite production, using lignocellulosic fibermats from the Biocomposites Group; use of ELO FAMES as a reactive diluent for solvent-free resin production.

Summary of the activities described in detail in the 2018/2019 report:

1. Manufacturing epoxidized plant oils and their derivatives:
 - EHO, ELO used for making biobased composites using hemp/wood fibermats
 - ECamO used to produce castor oil replacement polyol (COROL)
 - ECO used to produce hydroxyl propyl esters of the epoxidized fatty acids, as intermediates to make flame retardant polyol FRP.
 - ELO is also used in making fatty acid methyl esters of epoxides fatty acids, which further is used in manufacturing biobased composites without use of solvent
2. Further optimization of transesterification process leading to solvent-free production of 3rd generation polyol and scaled-up production for industry testing
3. Manufacturing of ELOME using optimized transesterification process.
4. Optimization and semi-pilot scale manufacturing of FRP
5. COROL process optimization and pilot scale manufacturing

Summary of the activities described in detail below for 2019/2020:

These relate especially to objective 2, the demonstration of how several related oleochemicals could be produced through intermediates from a single plant.

1. Development of alkanolamides, a desirable oleochemical
2. Development of a new type of lipid epoxy resin through the solvent-free production of a biobased curing agent

Development of alkanolamides

The alkanolamides that are produced from fatty acids or fatty acid methyl esters through the reaction with amines or aminoalcohols are used in many areas, including as raw material for polyol manufacturing, as non-ionic surfactants and in a cosmetics industry. A local opportunity was identified for the use of a novel alkanolamide derivative of various plant oils, in erosion control products, which are widely used in industrial operations, building sites, parking lots, roadways etc. The preliminary results below led to a new collaboration and funded project with a Calgary based chemical company (Engenium)

An attempt was made to develop canola oil (CO) and epoxidized canola oil (ECO) based diethanolamides using diethanolamine (DEA) without use of catalyst or solvents. The main aim from this work is to develop:

- a) high hydroxyl functional fatty amide diols (diethanolamide polyols) directly from canola oil via amidation reaction, that can be used in polyurethane manufacturing;
- b) bi-functional diethanolamides containing epoxy and hydroxyl groups from ECO via amidation reaction, that can be used as non-ionic surfactants, for example in biobased erosion and dust control products with a respective water soluble curing agents;
- c) ultra high hydroxyl functional diethanolamide polyols from ECO via amidation and epoxy ring opening reactions in one pot and one stage. Such products can be used in manufacturing polyurethanes or can be as a reactive intermediate to further produce epoxidized derivatives with terminal epoxy groups by reacting with, e.g., epichlorohydrin.

Schematics of the diethanolamide formation reaction is given in **Appendix I**.

It is clear from the mechanism shown above that the successful amidation reaction of the oil or its epoxides with DEA should yield to the formation of three moles of fatty amid diol, while one mole of glycerol is liberated.

Prior carrying the reaction, possible reaction products that can occur between CO and DEA were evaluated and the calculation of the functional reactive groups has been made using the fatty acid profile of the CO. Table 1 (**Appendix II**) summarizes the calculated hydroxyl values and epoxy content of the resulting from reaction of CO and ECO with DEA. Note that in each scenario, the concentration of the respective fatty acids are normalized taking into account the change of the molecular weights of individual components due to reaction with DEA.

The canola oil used is comprised mainly of 64% oleic acid, 19% linoleic acid and 7% linolenic acids, and small amounts of saturated fatty acids. Complete epoxidation of CO leads to the formation of ECO with the total oxirane oxygen content (OOC) of 6.4%.

Amidation of CO with DEA should form a product with hydroxyl value (OHV) of about 305 mg KOH/g, while OHV of the amidified ECO is about 289 mg KOH/g. The reduced OHV for the amidified ECO products are resulted from the difference in MW of the ECO compared to CO. In addition, if the epoxy groups of the ECO is not reacted with the DEA, then the amidified ECO products should have OOC of 5.2%. However, in case of the epoxy group of the ECO is reacted with DEA, then the formed amidified/hydroxylated products (polyol) should yield OHV of 622 mg KOH/g (Table 1, **Appendix II**). Note that these calculations are carried out with an assumption a complete epoxidation, amidation or epoxy ring opening reactions occurred. In case of partial epoxidation, amidation or ring opening reactions, the final OOC or OHV will vary.

For example, an incomplete amidation reaction could yield to the formation of diacylglycerol (DAG) or monoacylglycerol (MAG).

Experimental: The experiments to make diethanolamides were carried out using CO and ECO at various temperatures. For comparative analysis, similar reactions were carried out using saturated triacylglycerol (TAG), tristearin (SSS), and an epoxidized linseed oil (ELO), with high epoxy content compared to ECO. The reaction process was monitored by GPC through the disappearance of the TAG peak for CO, ECO, SSS or ELO. In addition, FTIR is used to monitor the progress of the reaction by following the structural changes – via monitoring the changes in ester bonds, epoxy and hydroxyl groups. Table 2 in **Appendix III** highlights the measured values of OHV and OOC of the products after amidation processes of the selected CO, ECO and ELO with DEA at different reaction conditions. It should be noted that the mixture of all used reactants are immiscible with DEA and therefore forms turbid, non-transparent mixture. However, as amidation reaction progresses the mixture becomes completely transparent.

Amidation of SSS and CO with DEA at 130 °C. As can be seen from **Appendix III** Table 2, the amidation reaction of all starting lipids with DEA occurs without any catalyst. The amidation process for saturated (SSS) and unsaturated (CO) TAG under the conditions used, are quite long. Although the reaction of SSS and CO was carried out up to 14 h and 12 h, the major amidation reactions occur within 9 h and 7 h, respectively, where the reaction mixture turned to transparent. SSS based amidified products appears to be solid at room temperature, therefore the properties of this product was not measured.

As an example, the selected GPC chromatograms (for CO and 4h, 6h, 7h, 8h of reaction) in the Figure 1(A) (**Appendix IV**) demonstrates the structural changes occurring at different stages of the amidation reaction of the CO with DEA. The numbers above the respective curves indicates the sampling (reaction) time. Note that the GPC measurements were performed on the reaction products without clean-up of unreacted DEA and liberated glycerol.

The peak with the retention time (RT) of 5.70 min shows the CO TAG decrease over reaction period, while formation of the DAG and anticipated product of diethanolamides of the canola fatty acids (CODEAm) with RT of 5.95 min and 6.36 min respectively, can be seen. Note that due to the similar MW of the MAG (i.g. 356 g/mol for 2-monoolein) and the average MW of the CODEAm (368 g/mol, calculated from FA profile of CO) it is difficult to separate these 2 products in GPC chromatograms. Although the CO+DEA mixture turned transparent after 7 hours of reaction, the complete disappearance of CO TAG structure was not measured until after 8 h of reaction. An estimation of the abundance of reaction products using the peak areas from GPC chromatograms (Figure 1(B) (**Appendix IV**)), with an assumption the response factor of all products are similar. The results showed that after ~8 hours of reaction, the TAG structure of the CO completely disappears, and with the formation of 13% DAG and 87% of CODEAm+MAG. Negligible change is observed in the products upon further mixing up to 12 hours.

Formation of the CODEAm was also checked by measuring the hydroxyl value of the products after cleaning-up the samples in THF using saturated NaCl solution. About 25 g of sample was dissolved in about 25 ml of THF in a separating funnel. Then, about 40 ml of NaCl solution was added and the solution is mixed well. The aqueous fraction containing glycerol and unreacted DEA was drained out. This procedure was repeated 3 times and, the traces of water removed using anhydrous sodium sulfate, and THF removed by using rotary evaporator at reduced pressure. The final product, CODEAm, had a yellow color and had a viscosity of 0.7 Pa.s, as

measured by a rheometer. The hydroxyl value of the CODEAm was measured according to ASTM standards (ASTM E222-17) and appears to be 254.2 ± 6.3 mg KOH/g (Table 2, **Appendix III**). This OHV is about 20% smaller than the calculated value and this was thought to be due to an incomplete amidation reaction. This hypothesis was confirmed by GPC chromatograms, which show that the product still contains about 13% of DAG, and by FTIR, which demonstrates the remaining presence of some of the original ester carbonyl band at 1743 cm^{-1} (Figure 1(C)). Figure 1(C) (**Appendix IV**) demonstrates the ATR-FTIR spectra of CO, DEA and selected CO+DEA reaction mixture (4h, 6h, 7h and 8h) upon amidation reaction. Note that the FTIR measurements were performed on the reaction products without clean-up of unreacted DEA and liberated glycerol. It is seen that DEA results in broad peaks at between $3000\text{-}3600\text{ cm}^{-1}$, which includes alcohol vibrations along with secondary amine bending (3300 cm^{-1}). DEA also gives strong vibration bands (C-O) at 1046 cm^{-1} . CO shows an ester carbonyl band (C=O stretching mode) at 1743 cm^{-1} and original C-O bands at 1160 cm^{-1} . The amidation reaction lead to the reduction of these ester carbonyl band and C-O bands, along with formation of the alcohol groups at between $3000\text{-}3600\text{ cm}^{-1}$, amide carbonyl group at 1618 cm^{-1} and the formation of C-O bands at 1046 cm^{-1} over the reaction period. The relatively slow amidation reaction of the CO (as well as SSS) with DEA is interpreted as due to the non-polar nature of the above-mentioned TAGs.

Amidation of ECO with DEA at 130 °C. The amidation reaction of ECO with DEA to produce diethanolamides of the epoxidized canola fatty acids (ECODEAm) was carried out at two different temperatures, at 130 °C and 110 °C , under identical reaction conditions. **Appendix V** Figure 2 represents GPC chromatograms of the ECO+DEA system upon amidation reaction at 130 °C (A), the abundance of the respective reaction products (B) calculated from the relative areas and the ATR-FTIR spectra of the ECO+DEA system upon amidation reaction (C). As can be seen from Figure 2(A) (**Appendix V**), the amidation reaction occurs extremely fast, and the disappearance of the ECO TAG structure is observed as fast as within 1.5 hours of reaction, although the system turned to transparent after 70 min of reaction (Table 2). This fast reaction of ECO with DEA is ascribed as due to relatively polar structure of ECO compared to the CO or SSS, and possibly due to presence of some hydroxyl moieties formed during epoxidation process of CO. As can be seen from Figure 2(B) (**Appendix V**), the trend in the formation of the ECODEAm is similar to the canola oil based counterparts (**Appendix IV**), but at a much faster rate. After 2 hours of reaction, the ECO TAG structure had disappeared while the amount of DAG and anticipated ECODEAm+MAG products increased significantly. The ratio of DAG to ECODEAm+MAG is similar to that of CO based diethanolamides.

The oxirane oxygen content of the input ECO was about 5.68% (Table 2, **Appendix III**) while the calculated OOC values for the ECODEAm is about 5.17% (Table 1, **Appendix II**). However, the measured OOC content of the ECODEAm is 4.84%, which is slightly smaller than the anticipated values. This could be explained by the reaction of the epoxy group of the ECO with the amine group of the DEA to add a hydroxyl functionality to the system. The epoxy ring opening by the hydroxyl group can also be expected, however this type of reactions requires catalytic conditions. The calculated OHV for the ECODEAm was about 290 mg KOH/g, while the measured value is relatively high (345 mg KOH/g). This is also an indication of the occurrence of a degree of epoxy ring opening by the DEA to form additional hydroxyl functionality.

Figure 2(C) (**Appendix V**) demonstrates ATR-FTIR spectra of the ECO+DEA system during the amidation reaction. The FTIR spectra of the ECO system is similar to that with CO, although ECO does not show the peak at 3005 cm^{-1} responsible for the C=C bond vibration. However, ECO shows a weak peak at 826 cm^{-1} corresponding to C-O-C stretching from an oxirane vibration. This peak intensity decreases somewhat over the amidation reaction, which indicates to the reduction of the epoxy group abundance due to the reaction with DEA. This confirms the occurrence of some epoxy ring opening reaction during the amidation process for ECO, under these experimental conditions. Interestingly, despite achieving such a high hydroxyl functionality, the viscosity of ECODEAm is only about 2.1 Pa.s, which makes it well suited for polyurethane applications.

Amidation of ECO with DEA at 110 °C. In this experiment, the reaction of ECO with DEA is carried out at a reduced temperature of 110 °C. Figure 3 (**Appendix VI**) shows GPC chromatograms of the ECO+DEA system over the amidation reaction (A), the abundance of the respective reaction products calculated from the relative areas (B), and the ATR-FTIR spectra of the ECO+DEA system upon amidation (C). It is clear that the amidation reaction at 110 °C occurs relatively slowly compared to the process carried out at 130 °C (Figure 3(A)). The mixture turned to transparent after 3 hours of mixing (Table 2), however, complete disappearance of the ECO TAG structure is observed after 4 hours of reaction (Figure 3(A)).

As can be seen from Figure 3(B) (**Appendix VI**), no significant changes on product yields were observed after 5 hours of mixing. The final ECODEAm product yield produced at 110 °C was slightly higher compared to that produced at 130 °C. The DAG content of the product was about 12% and ECODEAm+MAG content was 88%, however, the measured OOC content of the ECODEAm produced at different temperatures were similar (4.84%). The measured OHV of the was slightly higher (385 mg KOH/g) compared to the product produced at 130 °C, possibly due due to a higher level of amidation of the product. The viscosity of this product is about 1.7 Pa.s, which is still well-suited for polyurethane applications.

The trend in FTIR spectra and the changes to the products occurred during amidation process at 110 °C is similar to that of produced at 130 °C. This can be seen in the change of the ester carbonyl band (C=O stretching mode) at 1743 cm^{-1} and original C-O bands at 1160 cm^{-1} as well as in the formation of amide carbonyl group at 1618 cm^{-1} and the formation of C-O bands at 1046 cm^{-1} over the reaction period.

Thus, it can be concluded that the temperature of the amidation reaction did not greatly affect the yield of the ECODEAm products, although the reaction occurs faster at higher temperature. The degree of loss of OOC was found to be similar at both temperatures.

Amidation of ECO with an excess amount of DEA at 180 °C. In the previous experiments we have used the 1 mole of the TAG (either CO or ECO) while the amount of the DEA was kept constant at 3 moles. In this experiment, we have used 1 mole of ECO TAG and 6.7 moles of the DEA with an expectation that 3 moles of DEA will react with ECO TAG to form ECODEAm, while 3.7 moles of the DEA will react with epoxy group of the product to yield an additional hydroxyl functionality. In an ideal scenario, if all of the intended reactions will occur then we should produce an amidified/hydroxylated product (polyol) with an average hydroxyl number of 620 mg KOH/g (Table 1, **Appendix II**).

As we have seen that the small change in the temperature did not affect much to the reaction of epoxy group with the DEA, therefore, in this experiment the temperature of the reaction was set to 180 °C to promote epoxy ring opening reactions.

As can be seen from Figure 4(A) (**Appendix VII**) that the reaction of ECO with DEA started as fast as during equilibration of temperature at 180 °C (noted as 0m), with the reduction of ECO TAG intensity and formation of DAG. The ECO+DEA system become transparent after 7 min of reaction and no traces of the TAG is observed after 30 min of reaction at 180 °C. However, the color of the mixture become darker (dark brown) over reaction period and the reaction was stopped after 60 min of mixing. It should be noted that the DAG content of this product was about 3%, while the low molecular weight fraction of this product, which includes MAG, ECODEAm and hydroxylated ECODEAm, was about 97%. The OOC content of the product was 3.3%, after 1 hour of reaction that is slightly lower than the OOC of the products produced at 110 and 130 °C. As expected, the hydroxyl number of this product was 395 mg KOH/g, while viscosity of this product is significantly higher (6.5 Pa.s) compared to the other produced products. Probably, a fully hydroxylated canola based diethanolamide polyol could be produced, if the reaction was kept longer to complete epoxy ring opening. However, the high viscosity and dark color of the product could be an issue in funding right application for this product. Therefore, at this stage this experiment requires more optimization, in order to balance the properties of final products such the OHV, the viscosity and color.

Amidation of ELO with DEA at 110 °C. For comparative analysis, we have produced amidified product using epoxidized linseed oil (ELO), which has significantly high epoxy content compared to ECO. The molar ratio of ELO TAG to DEA in the mixture was 1 : 3. The main goal of this experiment was to follow the change of the OOC during amidation reaction. The amidified fatty acids of the ELO (ELODEAm) could be a good candidate for making erosion control products with the use of water soluble curing agents, if the ELODEAm solution/suspension creates stable aqueous mixture.

The ELO+DEA mixture become transparent after 60 min of mixing and, did not show any traces of the ELO TAG (Table 2, **Appendix III**). The reaction was stopped after 100 min of mixing. At this time, the proportion of DAG fraction was about 10%, while the low MW fraction of ELODEAm+MAG was about 90%, a higher low MW fraction compared to ECODEAm, produced at the same temperature. The initial OOC of the product is reduced from 8.78% to 6.58%, after 100 min of reaction. The hydroxyl number of the ELODEAm was 415 mg KOH/g, while the viscosity was 4.3 Pa.s (Table 2, **Appendix III**). FTIR spectra of this system and the trend in the change in FTIR spectra upon amidation is similar to that of ECODEAm.

Concluding remarks and outlook:

Upon analysis of the results of the experiments above it can be concluded that:

- The formation of the diethanolamides using DEA occurs faster in the epoxidized TAG (e.g. ECO or ELO) compared to unsaturated (CO) and saturated (SSS) TAGs;
- The higher epoxy content the faster amidation reaction with DEA;
- Unsaturated TAG (e.g. CO) undergoes to amidation with DEA faster than that of saturated TAG (e.g. SSS);
- High temperatures facilitate faster amidation reaction;
- The amidation reaction is faster than epoxy ring opening under similar conditions;

- Epoxy ring opening reaction during amidation process requires optimization: this possibly can be accelerated if desired, by catalyst addition after completion of amidation;
- Preliminary analysis of the emulsion forming capabilities of the products demonstrated that the ECODEAm products are more stable emulsions than other products produced;
- These products have considerable potential in a variety of applications, but these still need to be evaluated systematically. This is currently underway for their application in erosion control products (Alberta Innovates project with Engenium, Calgary, AB).

Development of a new type of lipid epoxy resin through the solvent-free production of a biobased curing agent

Background

Previously, LCG has developed plant oil epoxide based resins, especially for use in composite materials. Such thermosetting resins can be used to produce fibre- or- particle- reinforced boards for use in construction or automotive panels, or used in coatings or as solid materials. We filed an invention record and pursued a patent application (USPP 14/442,458; Aldehyde Free Thermoset Bioresins and Biocomposites) on technology. Ultimately, this provisional patent was dropped, in part because of some practical constraints which became evident during manufacturing trials, both in an auto-parts facility and in preparation of boards for house siding. Although we successfully made composite boards with suitable mechanical properties for these applications using that resin technology, it required the use of significant amounts of solvent in preparing the pre-polymer, and in order to lower the viscosity of the resin to ensure sufficient penetration into the fibre-mat or bulk fibre. This made manufacturing difficult and was not considered acceptable by the companies we worked with.

From this previous work, it became apparent that there exists a need for a biobased resin system comprised of a biobased curing agent that is miscible with epoxidised vegetable oil, so as to overcome the need for solvent use in forming the prepolymer. Furthermore, the viscosity of the resin, which should have a long pot life a room temperature, should ideally be low enough to spray or paint onto a surface without solvent thinning. It was also seen as highly desirable that the resin system can be cured in ways which can be adapted to suit the application.

Working with plant oil epoxy resins produced within this project, a graduate student (V.Patel) and the PI have developed a new fully biobased curing agent, recently filed an invention record with TEC Edmonton “A novel biobased hardener for epoxy resins with solvent-free production and low temperature curing” (TEC Edmonton, ROI number 2020018). We have recently shown that the novel curing agents described, which are produced via a low-cost solvent-free synthesis, can be used directly with plant-oil-epoxides without the need for co-solvents to promote mixing. This opens up the use of the plant oil epoxides and their derivatives, including those produced within this project, to many new epoxy resin applications.

Hence, the new curing agent described within this ROI, along with the development of ELO FAME derivatives as reactive diluent described in the year 2 interim report, achieve the objectives of solvent free resin, and are more compatible with industrial composite manufacturing. With these technologies and the pilot scale capabilities directly developed within this project, we have attracted a new start-up company (ZilaWorks) to locate to Alberta, to develop a range of hemp-fibre and bioresin based products.

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5. Literature cited

Provide complete reference information for all literature cited throughout the report.

- [1]. J.M. Curtis, G. Liu, T.S. Omonov, E. Kharraz, "Polyol Synthesis from Fatty Acids and Oils", U.S. Patent No: US 9,216,940 B2, December 22, 2015.
- [2]. J.M. Curtis, T.S. Omonov, E. Kharraz, X. Kong, M.H. Tavassoli-Kafrani. Method for Polyol Synthesis from Triacylglyceride Oils, U.S. Patent 10,189,761 B2 Jan 29, 2019.
- [3]. T.S. Omonov and J.M. Curtis; Chapter 7. Plant Oil-Based Epoxy Intermediates for Polymers, pp 99-125. in: Bio-Based Plant Oil Polymers and Composites, Eds: S. Madbouly, C. Zhang, and M.R. Kessler, Elsevier (2016).
- [4]. P.B.E. McVetty, E. Mietkiewska, T.S. Omonov, J.M. Curtis, D. Taylor, R. Weselake, Chapter 5. Brassica spp. Oils, pp 113-156 in: Industrial Oil Crops, Eds: T. McKeon, D. Hildebrand, R. Weselake, D. Hayes; Academic Press/AOCS (2016).
- [5]. T.S. Omonov, J.M. Curtis. Aldehyde Free Thermoset Bioresins and Biocomposites, U.S. Patent Application 2016/0215088 A1, Jul. 28, 2016.
- [6]. S.K. Yeong, Z. Idris, H. Abu Hassan. Chapter 20. Palm Oleochemicals in Non-Food Applications, pp 587-624 in: Palm Oil - Production, Processing, Characterization, and Uses, Eds: Q.-M. Lai, C.-P. Tan, C.C. Akoh, AOCS Press (2012).
- [7]. T.S. Omonov, J.M. Curtis, "Biobased epoxy resin from canola oil" J. Appl. Polym. Sci. 2014, 131(8), 40142
- [8]. J.M. Curtis, T.S. Omonov, E. Kharraz. Synthesis of Polyols Suitable for Castor Oil Replacement, U.S. Patent 10,301,239 B2, May 28, 2019.

6. Project team (max ½ page)

Describe the contribution of each member of the R&D team to the functioning of the project. Also describe any changes to the team which occurred over the course of the project.

Prof. Jonathan Curtis: Principal investigator of the project.

Dr. Tolibjon Omonov: Research Associate, responsible for the planning, designing and conducting pilot experimental work and developing oleoderivatives. In addition, his contribution to the project includes gathering experimental results, mechanical and chemical; data acquisition; writing and contributing to reports.

Mr. V. Patel: PhD student, experimental work on functionalised plant oil derivatives, participated in composite manufacture and in the development of biobased resin curing agents

Mr Ereddad Kharraz: LCG laboratory manager, maintained pilot scale equipment, performed

pilot scale experiments and ran analytical equipment, coordinates lab activities and safety.

7. Benefits to the industry (max 1 page; respond to sections a) and b) separately)

- a) Describe the impact of the project results on the Alberta or western Canadian agriculture and food industry (results achieved and potential short-term, medium-term and long-term outcomes).
- b) Quantify the potential economic impact of the project results (*e.g.*, cost-benefit analysis, potential size of market, improvement in efficiency, etc.).

The growth of the oleochemical industry gives western Canadian oilseed producers strong opportunities to diversify their markets, and at the same time achieve environmental goals by promoting the use of biobased materials and chemicals. Throughout this project, LCG has worked with multiple industries and achieved significant technical success in their applications. Although no short-term commercial success has yet been achieved, the technologies developed for transforming plant oils into oleo chemicals and materials, are now further advanced and continue to attract attention and new projects, so the potential for longer term benefits remain high.

1. Canola oil, being the dominant oil produced in western Canada, is also the most important for commercialisation of oleochemicals due to price and availability. Canola oil was therefore the main focus for polyol development. 3GP polyols, could have many uses in polyurethanes and beyond, due to their high functionality, uniform structure and the adaptability of the process allowing for multiple variations. The 3GP process allows for a range of other biobased chemicals to be derived from each step, either directly or as a derivative, so that a single 3GP plant would be able to manufacture a range of oleochemicals. In the earlier part of this project, LCG was working with a start-up venture OleoFoam, to develop rigid foam insulation for construction. Scale-up efforts were facilitated by this project and negotiations were made with Meadow Polymer, a subsidiary of Consolidated Biofuels (Delta BC) to manufacture commercial quantities of canola-based 3GP, utilizing knowledge gained through this project. Through technical association with LCG (Consolidated have licensed an earlier polyol version from UofA/LCG and have added further development of their own) Consolidated attracted a multi-million dollar investment from Sustainable Development Technology Canada to build a 6000tonne/year canola polyol plant. This project has directly contributed to that success by supporting research into pilot scale polyol developments thereby allowing us to provide technical support. In addition, the Consolidated plant, when complete, will provide new capability to the region for the scale-up of other oleochemicals. Although OleFoam no longer exists, we found interest in the PU foam product we developed from a company in St Albert, AB (Quantum Chemical). Through a MITACS postdoctoral fellow (now a permanent employee of that company), we were able to further develop the fire retardant properties of the foam. Multiple pilot scale batches of canola polyols were produced under this project for their evaluation.

2. Camelina, hemp and flax oils have niche markets which can be further developed through oleochemical industry taking advantage of their high iodine values Although a distinct technology (now patented by us), the previously described castor oil replacement polyol can be

produced as a variation on the 3GP production process. Originally based on camelina oil, but also successful using a blend of other local oils giving the fatty acid right profile, this could replace castor oil in many of its applications in the lubricant, polymer and personal care fields. A small inroad into this \$2bn+ market would be a substantial return for the Alberta economy. We have achieved the capability to make sufficient quantities for product testing.

3. Oleochemicals developed or further developed during this project include biobased epoxy resins and reactive amides. There is considerable interest in the area of plant oil epoxy resins, especially based on hemp since these could allow hemp fibre/hemp resin composite materials to be developed, as hemp production increases. Hemp or flax are especially suitable for these applications. Based on our developments in this area, we have attracted a company (ZILAWorks) who are setting up in the Edmonton area to create hemp- and other biobased materials, initially in the sporting goods field. They have already attracted private investment, are in the process of obtaining further grant funding and we have already applied for Mitacs funding to take the project further. A small start up based in Red Deer (Fetch Kayays/Earth Tone Promotional Products) is testing LCG hemp resin in hemp fibre biocomposite consumer products.

4. Another direct spin-off from this project is a collaboration with Engenium Chemicals (Calgary, AB) to develop lipid based erosion control products, with funding from Alberta Innovates. A combination of the fatty amide and lipid epoxy resin technologies discussed above has helped develop a prototype product now under investigation.

8. Contribution to training of highly qualified personnel (max ½ page)

Specify the number of highly qualified personnel (*e.g.*, students, post-doctoral fellows, technicians, research associates, etc.) who were trained over the course of the project.

- 1 Research Associate (Dr. Tolibjon Omonov) was involved in most of this research – this experience has directly led to follow-on industry related projects as described in Section 7.
- 1 PhD student (Vinay Patel) was trained and partly supported through this project. This project led to an application to MITACS for support for this student to do an industry internship as part of his PhD, working on lipid resins.
- 1 Laboratory Technician (Ereddad Kharraz) was trained in pilot production of the oleochemicals and supported through this project. This, and his other experience from this project directly benefits all future oleochemical projects and students.
- Dr M. Hossein Tavassoli Kafrani, a former LCG student and MITACS Accelerate postdoctoral fellow, received material support from this project in the form of pilot scale production of 3GP and other polyols, needed for his further development of fire-retardant PU foam products at Quantum Chemical. Dr Tavassoli Kafrani is now a full-time employee of Quantum Chemical, St Albert, AB.

9. Knowledge transfer/technology transfer/commercialisation (max 1 page)

Describe how the project results were communicated to the scientific community, to industry stakeholders, and to the general public. Please ensure that you include descriptive information, such as the date, location, etc. Organise according to the following categories as applicable:

- a) Scientific publications (e.g., scientific journals); attach copies of any publications as an appendix to this final report
- b) Industry-oriented publications (e.g., agribusiness trade press, popular press, etc.); attach copies of any publications as an appendix to this final report
- c) Scientific presentations (e.g., posters, talks, seminars, workshops, etc.); attach copies of any presentations as an appendix to this final report
- d) Industry-oriented presentations (e.g., posters, talks, seminars, workshops, etc.); attach copies of any presentations as an appendix to this final report
- e) Media activities (e.g., radio, television, internet, etc.)
- f) Any commercialisation activities or patents

N.B.: Any publications and/or presentations should acknowledge the contribution of each of the funders of the project, as per the investment agreement.

A. Related publications/submitted manuscripts:

1. The Development of Epoxidized Hemp Oil Prepolymers for the Preparation of Thermoset Networks

Tolibjon S. Omonov, Vinay Patel, , Jonathan M. Curtis
J. Am Oil Chem. Soc. 96, 1389-1403 (2019)

2. Epoxidized plant oil based natural fibre composites

Vinay Patel, Tolibjon S. Omonov, Cagri Ayranci, Jonathan M. Curtis
Submitted to J. Composite Materials 2019

3. Biobased thermosets from epoxidized linseed oil and its methyl esters

Tolibjon S. Omonov, Vinay Patel, Jonathan M. Curtis
Submitted to IC&P 2019

B. Presentations:

1. Omonov T.S., Patel V.R., Curtis. J.M., "Towards complete utilization of hemp products in composite materials", oral presentation at the *Pacific Rim Hemp Conference 2018*, July 18-19 2018, Vancouver, BC, Canada.
2. Omonov T.S., Patel V.R., Curtis. J.M., "Epoxidized hemp oil: Synthesis, cure kinetics and properties of polymers", Poster presentation at the *15th International Symposium on Bioplastics, Biocomposites and Biorefining*, July 24-27, 2018, Guelph, ON, Canada.
3. Patel V.R., Omonov T.S., Curtis. J.M., Ayranchi C., "Epoxidized plant oil based natural fiber composites", Oral presentation at the *15th International Symposium on Bioplastics, Biocomposites and Biorefining*, July 24-27, 2018, Guelph, ON, Canada.
4. Omonov T.S., Patel V.R., Curtis. J.M. "The use of epoxidized linseed oil and its methyl esters in thermoset resins", Oral presentation at the *26th Canadian Lipid & Bioresource Conference*, September 9-11, 2018, Saskatoon, SK, Canada.
5. Omonov T.S., Patel V.R., Curtis. J.M. "Hemp oil based bioresins: Cure kinetics and properties", Poster presentation at the *26th Canadian Lipid & Bioresource Conference*, September 9-11, 2018, Saskatoon, SK, Canada.

6. Curtis. J.M., Kong X., Omonov T.S., “Developing lipid-derived intermediates for the synthesis of high biocontent”, Oral presentation at the 101st *Canadian Chemistry Conference and Exhibition*, May 27-31, 2018, Edmonton, AB, Canada
7. Invited talk for American Oil Chemists Society 2020 meeting on “Advances in Lipid Based Epoxy Resins was cancelled due to the pandemic.

C. Patents utilised in this project, issued during this period but previously submitted:

1. Synthesis of Polyols Suitable for Castor Oil replacement (Curtis, Omonov, Kharraz)
U.S. Patent No. 10,301,239 issued May 28, 2019
2. Method for Polyol Synthesis from Triacylglyceride Oils (Curtis, Omonov, Kharraz, Kong, Tavassoli-Kafrani)
U.S. Patent No. 10,189,761 issued Jan.29, 2019

Section D: Project resources

1. Provide a detailed listing of all cash revenues to the project and expenditures of project cash funds in a separate document certified by the organisation’s accountant or other senior executive officer, as per the investment agreement. Revenues should be identified by funder, if applicable. Expenditures should be classified into the following categories: personnel; travel; capital assets; supplies; communication, dissemination and linkage (CDL); and overhead (if applicable).
2. Provide a justification of project expenditures and discuss any major variance (*i.e.*, ± 10%) from the budget approved by the funder(s).
3. Resources:
Provide a list of all external cash and in-kind resources which were contributed to the project

Total salary costs at \$351.9k were slightly above the budgeted \$327k amount, but the \$24k allocated costs for testing were absorbed into other related activities so compensate for this amount. The materials and supplies at \$30k matched the budgeted \$31k as did \$6k travel cost match the \$6k budget. Unfortunately the \$25k company cash funding did not materialise as noted earlier, but this was beyond our control.

Total resources contributed to the project		
Source	Amount	Percentage of total project cost
Agriculture Funding Consortium	\$388,000	72.73%
Other government sources: Cash	\$0	0.00%
Other government sources: In-kind	\$115,500	21.65%
Industry: Cash	\$0	0%
Industry: In-kind	\$30,000	5.62%
Total Project Cost	\$533,500	100%

External resources (additional rows may be added if necessary)		
Government sources		
Name (no abbreviations unless stated in Section A3)	Amount cash	Amount in-kind
University of Alberta	-	115,000

Industry sources		
Name (no abbreviations unless stated in Section A3)	Amount cash	Amount in-kind
BioFoam (1782815 Alberta Ltd) <i>(no longer in business)</i>	—	30,000

Section E: Research Team Signatures and Authorised Representative's Approval

The Principal Investigator and an authorised representative from the Principal Investigator's organisation of employment **MUST** sign this form.

Research team members and an authorised representative from their organisation(s) of employment **MUST** also sign this form.

By signing as an authorised representative of the Principal Investigator's employing organisation and/or the research team member's(s') employing organisation(s), the undersigned hereby acknowledge submission of the information contained in this final report to the funder(s).

Principal Investigator

Principal Investigator	
Name: Jonathan Curtis	Title/Organisation: Professor / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Signature: 	Date: 15th July 2020
Principal Investigator's Authorised Representative's Approval	
Name:	Title/Organisation:
Signature:	Date:

Research Team Members (add more tables as needed)

1. Team Member	
Name: Tolibjon Omonov	Title/Organisation: Research Associate / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Signature:	Date:
Team Member's Authorised Representative's Approval	
Name: Jonathan Curtis	Title/Organisation: Professor / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
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2. Team Member	
Name: Ereddad Kharraz	Title/Organisation: Laboratory Technician / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
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Name: Jonathan Curtis	Title/Organisation: Professor / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Signature:	Date:

3. Team Member	
Name: Vinay Patel	Title/Organisation: PhD Student / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
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Name:	Title/Organisation:

Jonathan Curtis	Professor / LCG, Department of Agricultural, Food and Nutritional Science, University of Alberta
Signature:	Date:

Section F: Suggested reviewers for the final report

Provide the names and contact information of four potential reviewers for this final report. The suggested reviewers should not be current collaborators. The Agriculture Funding Consortium reserves the right to choose other reviewers. Under *Section 34* of the *Freedom of Information and Protection Act (FOIP)* reviewers must be aware that their information is being collected and used for the purpose of the external review.

Reviewer #1

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